AD-A047 064 WEAPONS RESEARCH ESTABLISHMENT SALISBURY (AUSTRALIA) F/G 7/2
AN X-RAY FLUORESCENCE METHOD OF ANALYSING THIN FILMS OF INDIUM --ETC(U)
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UNCLASSIFIED WRE-TR-1770(A) NL

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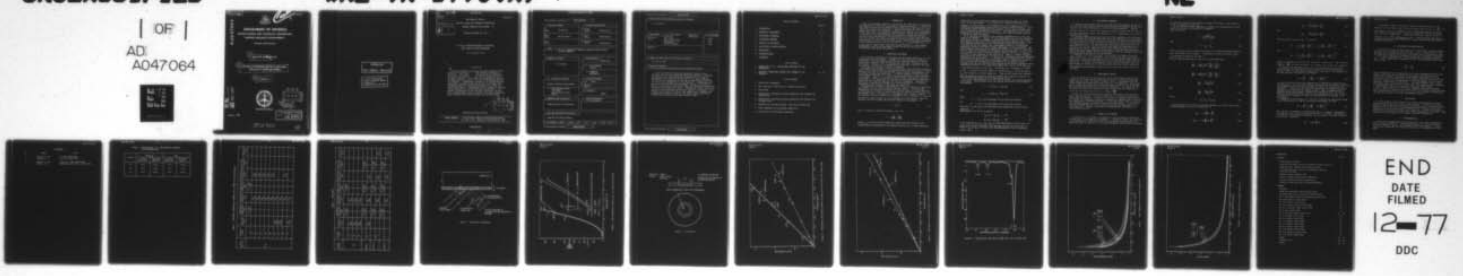

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(14) WRE-TR-1770 (A)

AR-000-529

AD A047064

AUSTRALIA

DEPARTMENT OF DEFENCE

DEFENCE SCIENCE AND TECHNOLOGY ORGANISATION

WEAPONS RESEARCH ESTABLISHMENT

SALISBURY, SOUTH AUSTRALIA

(9) TECHNICAL REPORT 1770 (A)

(6) AN X-RAY FLUORESCENCE METHOD OF ANALYSING THIN FILMS OF INDIUM ANTIMONIDE.

(10) R.H. HARTLEY

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TECHNICAL REPORT 1770 (A) ✓

AN X-RAY FLUORESCENCE METHOD OF ANALYSING THIN FILMS OF INDIUM ANTIMONIDE

R.H. Hartley Ph.D.

SUMMARY

A precision method of measuring the atomic composition and thickness of thin films containing indium and antimony is described. This method depends on measuring the attenuation by the film of fluorescent X-rays from the calcium atoms in the glass substrate. The results are independent of layering or compositional variations in the direction perpendicular to the substrate. Measurements are made at two calcium emission wavelengths, selected so that one of these lies between the L absorption edges for indium and antimony and the other lies outside that region. Experimental results confirm the validity of the technique, and an error analysis is presented. The method is applicable to films of other composition, including those containing more than two elements, for which examples are given, together with suitable fluorescing substrate atoms.

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POSTAL ADDRESS: The Director, Weapons Research Establishment, Box 2151, G.P.O., Adelaide, South Australia. 5001.

DOCUMENT CONTROL DATA SHEET

Security classification of this page: UNCLASSIFIED

1 DOCUMENT NUMBERS	
AR Number:	AR-000-529
Report Number:	WRE-TR-1770(A)
Other Numbers:	

2 SECURITY CLASSIFICATION	
a. Complete Document:	Unclassified
b. Title in Isolation:	Unclassified
c. Summary in Isolation:	Unclassified

3 TITLE: AN X-RAY FLUORESCENCE METHOD OF ANALYSING THIN FILMS OF INDIUM ANTIMONIDE

4 PERSONAL AUTHOR(S):

R.H. Hartley

5 DOCUMENT DATE: March 1977

6 6.1 TOTAL NUMBER OF PAGES: 23

6.2 NUMBER OF REFERENCES: 2

7 7.1 CORPORATE AUTHOR(S):

Weapons Research Establishment

7.2 DOCUMENT (WING) SERIES AND NUMBER

Applied Physics Wing
TR-1770

8 REFERENCE NUMBERS

a. Task: 70/05

b. Sponsoring Agency:

9 COST CODE: 228114

10 IMPRINT (Publishing establishment):

Weapons Research Establishment

11 COMPUTER PROGRAM(S) (Title(s) and language(s))

12 RELEASE LIMITATIONS (of the document):

Approved for Public Release

12.0	OVERSEAS	NO		P.R.	1	A		B		C		D		E	

Security classification of this page: UNCLASSIFIED

Security classification of this page: UNCLASSIFIED

13 ANNOUNCEMENT LIMITATIONS (of the information on these pages):

No limitation

14 DESCRIPTORS:

a. EJC Thesaurus Terms

X-ray fluorescence
Analyzing
Thin films
Indium antimonides
Measurement
Atomic structure
Attenuation
Substrates

b. Non-Thesaurus Terms

15 COSATI CODES:

2006
1402
0702

16 LIBRARY LOCATION CODES (for libraries listed in the distribution):

SW SR SD AACA

17 SUMMARY OR ABSTRACT:
(if this is security classified, the announcement of this report will be similarly classified)

A precision method of measuring the atomic composition and thickness of thin films containing indium and antimony is described. This method relies on measuring the attenuation by the film of fluorescent X-rays from the calcium atoms in the glass substrate. The results are independent of layering or compositional variations in the direction perpendicular to the substrate. Measurements are made at two calcium emission wavelengths, selected so that one of these lies between the L absorption edges for indium and antimony and the other lies outside that region. Experimental results confirm the validity of the technique, and an error analysis is presented. The method is applicable to films of other composition, including those containing more than two elements, for which examples are given, together with suitable fluorescing substrate atoms.

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1. INTRODUCTION

There is considerable interest in determining the total thickness and percentage composition of thin films consisting of indium and antimony. A method has been found for doing this by measuring the attenuation of characteristic x-rays emitted by calcium in the substrate. Indium and antimony are close in atomic number, hence their mass absorption coefficients are similar at all wavelengths except those between corresponding absorption edges, and for most wavelengths it is difficult to distinguish between the absorption of indium and antimony. A substrate containing calcium was chosen because one of the principal emission lines lies between the indium and antimony L absorption edges, where the attenuation produced by indium is significantly greater than that produced by antimony. Another calcium emission line lies outside the L_{In} - L_{Sb} wavelength band, i.e. in the spectral region where indium and antimony behave in a similar manner. Measurements at these two wavelengths enable the mass per unit area to be evaluated for both indium and antimony.

2. THEORETICAL BACKGROUND

It is desired to determine the thickness and percentage composition of a film containing indium and antimony. Assuming that the film whose thickness and composition is to be determined contains only indium and antimony, uniformly distributed throughout, then the film mass per unit area is constant over the test region. Finally, it is assumed that the substrate is perfectly flat and has calcium atoms uniformly distributed throughout.

The method to be discussed involves a comparison of the intensity of fluorescent calcium radiation from a clean substrate surface with the intensity obtained from the other side of the same substrate coated with the indium/antimony film of unknown thickness and composition. A similar technique has been used for single element films(ref.1), in which case measurements at a single wavelength will suffice. Where it is required to analyse films containing two elements, measurements at two wavelength are required, together with certain conditions to ensure that the resulting equations are linearly independent. Obviously the method may be extended to n element films where measurements at n different wavelengths would be needed to determine the composition.

The underlying principle of the method may be best illustrated by considering the simplest case of a film containing only one element. Thus Cline and Schwartz(ref.1) measured the thickness of aluminium films on silicon substrates by observing the intensity of the Kα emission line from the substrate both with and without a film. The geometrical arrangement used is shown in figure 1. If the uncoated substrate fluoresces at wavelength λ with an intensity I_o, then the presence of a film (in this case, an aluminium film) of thickness t (units of mass/unit area) will reduce the measured intensity at wavelength λ, to

$$I = I_o e^{-At} \tag{1}$$

Here, A is given by (Cline and Schwartz, loc. cit.)

$$A = \frac{\mu_p}{\sin\theta} + \frac{\mu_S}{\sin\phi} \tag{2}$$

where μ_p is an effective mass absorption coefficient which depends on the average effective wavelength of the primary radiation, μ_S is a mass absorption

coefficient for the fluorescence radiation and θ and ϕ are input and output angles of the spectrometer. It is difficult to evaluate the factor μ_p in equation (2) theoretically, but fortunately this is not required if A can be determined from measurements using a series of films of known thickness. Cline and Schwartz showed experimentally that equation (1) was valid, and they were able to determine an appropriate value for A. The technique was then used to determine the thickness of silicon films which for technical reasons could not be obtained by direct weighing.

Let us now apply this method to the thickness determination of a film composed of two elements. Clearly, at X-ray wavelengths where the mass absorption coefficients for the two elements are practically equal, only the total thickness (mass/unit area) but not the composition may be determined as above. But if in addition we were able to find a wavelength at which the absorption coefficients for the two elements are significantly different, the percentage composition could be found from a further measurement at this wavelength. The mass absorption curves(ref.2) for indium and antimony (see figure 2), show that for most values of wavelength the attenuation of an indium film is close to that of an antimony film of the same area density, making it difficult to separate the effect of each component in the binary film. However, the mass absorption coefficients for certain values of λ between the L absorption edges are appreciably different for the two elements. In particular, the wavelength 3.089 Å (Ca Kβ_1) lies above the L absorption edge for antimony, where μ = 300 cm^2/gm, whereas it lies in the transition region for indium where μ is considerably higher. Although values for this portion of the spectrum are not given in standard tables, experiments described in this memorandum suggest that, for the Ca Kβ line, μ_{In} is approximately equal to twice μ_{Sb}. By contrast at a wavelength of 3.360 Å (Ca K$\alpha_{1,2}$), neither transition region is involved and μ_{In} = 310 cm^2/gm, whilst μ_{Sb} (365 cm^2/gm) is of similar magnitude. Thus calcium fluorescence radiation from the supporting substrate should be suitable for a complete evaluation of both thickness and composition of indium/antimony films.

Let us now extend equation (1) to the case of a binary film containing indium and antimony. Corresponding to the two calcium fluorescence lines we obtain two equations:

$$a = e^{-(A_{In}\, t_{In} + A_{Sb}\, t_{Sb})} \tag{3}$$

and

$$a' = e^{-(A'_{In}\, t_{In} + A'_{Sb}\, t_{Sb})} \tag{4}$$

where

a = I/I_o at the wavelength of the calcium Kα emission

and

a' = I/I_o at the wavelength of the calcium Kβ emission,

and finally t_{In} and t_{Sb} are the equivalent mass thicknesses of the two film components.

From equations (3) and (4) we obtain:

$$A_{In}\, t_{In} + A_{Sb}\, t_{Sb} = -\ln a \tag{5}$$

$$A'_{In}\, t_{In} + A'_{Sb}\, t_{Sb} = -\ln a' \tag{6}$$

If the constants A_{In}, A'_{In}, A_{Sb}, A'_{Sb} are found experimentally using single component films of known thickness, then equations (5) and (6) may be used to evaluate t_{In} and t_{Sb} for binary films from measurements of the 'attenuation' values a and a'.

3. EXPERIMENTAL PROCEDURE

To determine experimentally the four constants used in equations (5) and (6), two series of single element films of known thickness were produced and the ratio I/I_o for every film was measured for the two calcium wavelengths. Semilogarithmic plots of I/I_o versus film thickness yielded a straight line for each of the four experimental series, thus permitting the 'A' coefficients to be derived from the line slopes.

In preparing indium and antimony films for these experiments, care was taken to ensure films were of uniform thickness across the substrate of 2.2 cm diameter. Thus to reduce the thickness variations to about 1%, the films were deposited in a vacuum on soda lime glass substrates which were held a distance of 38 cm from the evaporant source. The deposition was carried out slowly at rates of the order of 10 Å/s or less and the substrates were rotated at approximately half a revolution per second. Substrates were weighed before and after the film deposition, and an edge clamping device was used to hold the substrate without masking the front surface, so that surface density could be obtained from the two mass measurements and the area of the substrate.

To expose a film to the x-ray beam, it was placed in a holder of the type shown in figure 3. It is desirable to use a holder which does not produce fluorescence radiation at wavelengths close to the calcium $K\alpha$ and $K\beta_1$ emissions. Satisfactory results were obtained with a heavily gold plated holder machined from brass. A central area of 0.86 cm^2 on each film was exposed to the x-ray beam through the opening in the substrate holder and I and I_o were determined from appropriate measurements using the film side and the reverse side of the substrate.

4. EXPERIMENTAL RESULTS

The 'A' coefficients of equations (5) and (6) were obtained from the experimental data on single element indium and antimony films ignoring background counts and the results are shown in figures 4 and 5. The presence of interfering indium and antimony fluorescence peaks was also neglected. This latter point is mentioned particularly, because as shown in figure 6, the indium peak may contribute appreciably to the calcium $K\alpha$ background. The values for the absorption constants were obtained by using a least squares fit which would force the line through the origin (i.e. slope of $\frac{\Sigma xy}{\Sigma x^2}$).

Finally, the four coefficients thus evaluated were used to calculate from X.R.F. data the thickness and atomic composition of several films containing both indium and antimony. In Table 1 the results are compared with estimates of thickness and composition obtained by weighing the substrates before and after each deposition of indium and antimony. As the table shows, there is satisfactory agreement between the results yielded by the two methods. It may therefore be concluded that with the use of our experimentally determined 'A' coefficients X.R.F. affords a ready means for assessing the stoichiometry of Indium antimonide films.

5. ACCURACY OF THE METHOD

The method can, in principle, be applied over the full range of composition to films up to several mgm/cm^2 in thickness. In practical terms, the limitation of the method arises from counting errors, particularly for thick films, for which the signal is reduced to a level where background counts become significant.

We shall carry out our error analysis in terms of the errors in the quantities a and a' of equation (3) and (4).

Let the fractional atomic component of indium in an indium/antimony film be p and let the total film thickness be T(mgm/cm^2).
Then we can write:

$$p = \frac{t_{In}/a}{t_{In}/a + t_{Sb}/b} \tag{7}$$

and

$$T = t_{In} + t_{Sb} \tag{8}$$

where t_{In} and t_{Sb} are the 'partial thicknesses' of indium and antimony (mgm/cm^2) and a and b their atomic weights. In equations (5) and (6) we have t_{In} and t_{Sb} expressed as functions of a and a'.
Using in addition equations (7) and (8) it can be shown that:

$$\frac{\partial p}{\partial a} = \frac{-p(1 - p)}{a\ D}\left[\frac{A'_{Sb}}{t_{In}} + \frac{A'_{In}}{t_{Sb}}\right] \tag{9}$$

$$\frac{\partial p}{\partial a'} = \frac{p(1 - p)}{a'\ D}\left[\frac{A_{Sb}}{t_{In}} + \frac{A_{In}}{t_{Sb}}\right] \tag{10}$$

$$\frac{\partial T}{\partial a} = \frac{A'_{In} - A'_{Sb}}{a\ D} \tag{11}$$

and

$$\frac{\partial T}{\partial a'} = \frac{A_{Sb} - A_{In}}{a'\ D} \tag{12}$$

where

$$D = A_{In}\ A'_{Sb} - A'_{In}\ A_{Sb} \tag{13}$$

From equations (9) to (13) one finds the variations in p and T due to given fractional changes in a and a' respectively as

$$\delta p_a = \left(a \frac{\partial p}{\partial a}\right) \times \left(\frac{\delta a}{a}\right) \tag{14}$$

$$\delta p_{a'} = \left(a' \frac{\partial p}{\partial a'}\right) \times \left(\frac{\delta a'}{a'}\right) \tag{15}$$

$$\delta T_a = \frac{A'_{In} - A'_{Sb}}{D} \times \left(\frac{\delta a}{a}\right) \tag{16}$$

and

$$\delta T_{a'} = \frac{A_{Sb} - A_{In}}{D} \times \left(\frac{\delta a'}{a'}\right) \tag{17}$$

The overall precision in p and T is given by:

$$\Delta p = \left\{ \left[\left(a \frac{\partial p}{\partial a}\right) \times \left(\frac{\delta a}{a}\right) \right]^2 + \left[\left(a' \frac{\partial p}{\partial a'}\right) \times \left(\frac{\delta a'}{a'}\right) \right]^2 \right\}^{\frac{1}{2}} \tag{18}$$

and

$$\Delta T = \left\{ \left[\left(a \frac{\partial T}{\partial a}\right) \times \left(\frac{\delta a}{a}\right) \right]^2 + \left[\left(a' \frac{\partial T}{\partial a'}\right) \times \left(\frac{\delta a'}{a'}\right) \right]^2 \right\}^{\frac{1}{2}} \tag{19}$$

which are dependent on the errors $\delta a'$ in a and a' respectively. The quantities $a \frac{\partial p}{\partial a}$ and $a' \frac{\partial p}{\partial a'}$ are shown in figure 7 as a function of T using p as a parameter.

In general, the experimental error in a will be different from that in a', since different counting rates may be associated with the two measurements. If however we make the simplifying assumption that both a and a' are determined to the same accuracy, then equation (18) becomes

$$\Delta p = \left[\frac{\delta a}{a} \left(a \frac{\partial p}{\partial a}\right)^2 + \left(a' \frac{\partial p}{\partial a'}\right)^2 \right]^{\frac{1}{2}} \tag{20}$$

i.e. the error in the fractional composition p is obtained by multiplying the fractional error in a by an "error factor" represented by the radical in equation (20), numerical values for which are shown in figure 8 as a function of the thickness T. Whilst at first sight this figure seems to indicate that higher accuracy in p is attainable for thicker films, it must be remembered that a greater film thickness implies increased x-ray absorption, which at constant intensity of the incident beam leads to lower counting rates, so that a rise in the statistical uncertainty in a may well counterbalance the decrease in the error factor.

Turning now to the accuracy in T, and still retaining our simplifying assumption of equal errors in a and a', we obtain from equation (19) that

$$\Delta T = \frac{\delta a}{a} \left[\left(a \frac{\partial T}{\partial a}\right)^2 + \left(a' \frac{\partial T}{\partial a'}\right)^2 \right]^{\frac{1}{2}} \tag{21}$$

This equation is analogous to equation (20), but it is apparent from equations (11) and (12) that in this case the error factor is a constant, independent of T and from the experimentally determined 'A' coefficients its value is found to be 0.674 μm. Therefore

$$\Delta T = 0.674 \frac{\delta a}{a} \ \mu m \tag{21a}$$

As a numerical example of the use of equations (20) and (21a), let the fractional error in both a and a' have the typical value of 0.1%, i.e. $\frac{\delta a}{a} = 10^{-3}$ and consider a film of 0.5 mgm/cm^2 thickness containing about 50% of indium. From figure 8 the error factor is then 2.3, so that $\Delta p = 0.0023$, implying an error of 0.23% in composition. Similarly from equation (21a) the error in thickness is $\pm$ 6.7 Å or approximately 0.07%. Clearly therefore the method is suitable for high precision determination of both thickness and composition of indium/antimony films.

6. APPLICATION TO OTHER MATERIALS

So far the discussion has been restricted to In/Sb films, but the technique can obviously be extended to other materials. This is merely subject to the constraint that substrate fluorescence wavelengths must be available for which the equations corresponding to equations (5) and (6) are linearly independent.

It is readily shown that for a two component film, containing materials x and y, linear independence is guaranteed if their mass absorption coefficients are such that

$$\frac{\mu_x}{\mu'_x} \neq \frac{\mu_y}{\mu'_y} \qquad (22)$$

This inequality must not be understood in the strict mathematical meaning, but rather in the sense that the ratios on both sides of the expression are sufficiently different in magnitude for meaningful computations to be made. In practice this will be the case, if the absorption edge for one material (and only one) separates the two wavelengths at which measurements are to be made, otherwise an approximate equality holds. For three component films, linear independence may be achieved by selecting three wavelengths which are separated by the absorption edges of two (and only two) of the three elements.

Table 2 shows examples of several multi-component substances of current practical interest, together with suitable sources of substrate fluorescence and their relevant emission lines, but the table is clearly capable of expansion as the need for the analysis of other film materials arises.

7. CONCLUSIONS

The atomic composition and area density of thin indium/antimony films have been determined by an X.R.F. method of high precision. Because this method depends on the attenuation of fluorescent x-radiation from the substrate passing through the film, the results are independent of any layering or variation of composition in the direction normal to the substrate. Finally, the method may be applied to the analysis of thin films of many compounds other than InSb. Some examples are listed, together with suitable substrate combinations.

8. ACKNOWLEDGEMENTS

I would like to acknowledge the assistance of Mr. D.R. Smoker in preparing films for these experiments. The patient efforts of Mr. W.A.R. MacFarlane during the somewhat tedious X-ray measurements were valued greatly. Finally, I would like to thank Mr. E.H. Hirsch for his help in the preparation of this Report.

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No.	Author	Title
1	Cline, J.E. and Schwarts, S.	"J. Electrochem Soc." Vol.114, p 605, 1967.
2	Jenkins, R. and Devries, J.L.	"Practical X-Ray Spectrometry" MacMillan and Co. Ltd., p 181, 1970.

TABLE 1. COMPARISON OF X.R.F. AND WEIGHING ESTIMATES OF FILM COMPOSITION

Film No.	Weighing		XRF	
	% indium composition	'Thickness' (mgm/cm^2)	% indium composition	'Thickness' (mgm/cm^2)
52	58.2	0.344	57.5	0.343
53	33.3	0.310	31.7	0.313
60	45.0	0.682	40.6	0.693

TABLE 2. SUBSTRATE FLUORESCENCE SOURCES FOR A NUMBER OF FILM MATERIALS

Film material	Substrate atoms	Wavelengths of absorption edges and useful emission lines (Å)								
		Emission line	Absorption edge		Emission line	Absorption edge		Emission line	Absorption edge	Emission line
Ga As			As K edge 1.045			Ga K edge 1.195				
	Ta				$L\gamma_1$ 1.138			$L\beta_2$ 1.285		
	Ir				$L\beta_1$ 1.158			$L\alpha_1$ 1.352		
	Pt				$L\beta_1$ 1.120			$L\alpha_1$ 1.313		
	Au				$L\beta_1$ 1.083			$L\alpha_1$ 1.277		
	Hg				$L\beta_1$ 1.049			$L\alpha_1$ 1.242		
	Pb	$L\beta_1$ 0.982			$L\alpha_1$ 1.175					
	Bi	$L\beta_1$ 0.952			$L\alpha_2$ 1.144					
	Ge				$K\beta_1$ 1.129			$K\alpha_1$ 1.255		
	Se	$K\beta_1$ 0.992			$K\alpha_1$ 1.105					

TABLE 2. (CONTINUED)

Film material	Substrate atoms	Wavelengths of absorption edges and useful emission lines (Å)						
		Emission line	Absorption edge	Emission line	Absorption edge	Emission line	Absorption edge	Emission line
InP			In K edge 0.444		P K edge 5.787			
	Sn	$K\beta_1$ 0.435		$K\alpha_1$ 0.491				
	Sb	$K\beta_1$ 0.417		$K\alpha_1$ 0.470				
	Te	$K\beta_1$ 0.400		$K\alpha_1$ 0.451				
GaAlAs			As K edge 1.045		Ga K edge 1.195		Al K edge 7.951	
	Au	$L\gamma_1$ 0.927		$L\beta_1$ 1.083		$L\alpha_1$ 1.277		
Pb Sn Te			Pb L edge 0.782		Te L edge 2.510		Sn L edge 2.778	
	Ba			$L\beta_2$ 2.404		$L\alpha_1$ 2.776		$L\alpha_2$ 2.785
Cd Hg Te			Hg L edge 0.979		Te L edge 2.856		Cd L edge 3.084	
	I			$L\beta_2$ 2.751		$L\beta_1$ 2.937		$L\alpha_1$ 3.148

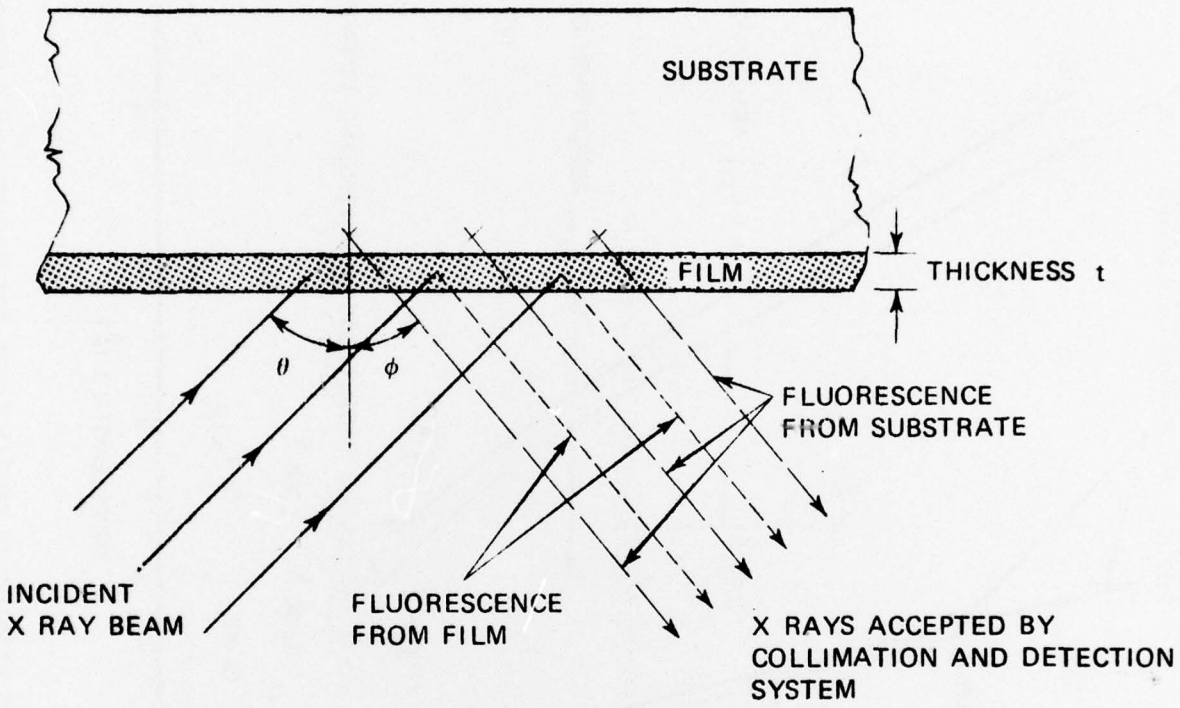

Figure 1. Geometrical arrangement

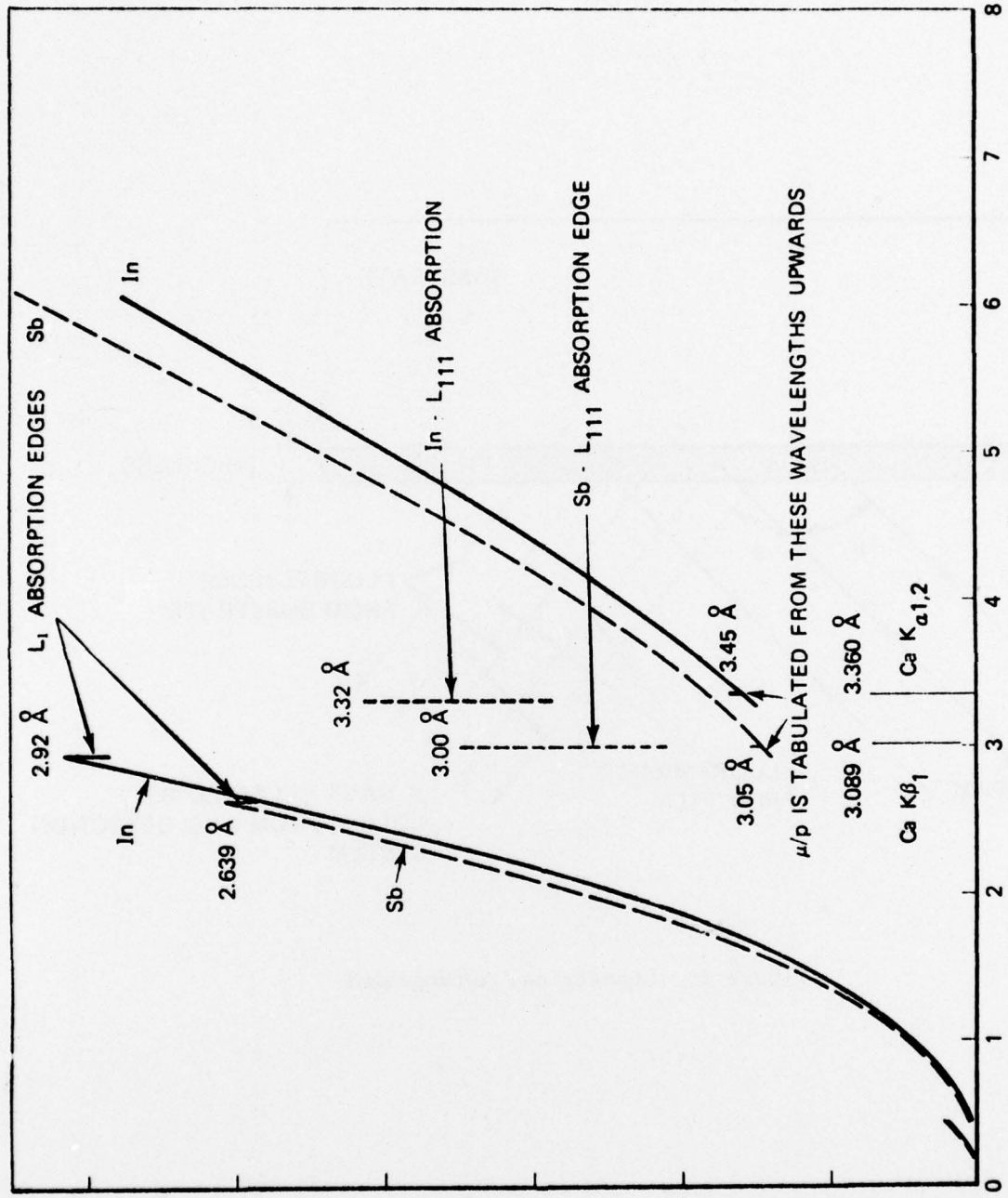

Figure 2. Mass absorption coefficients for indium and antimony

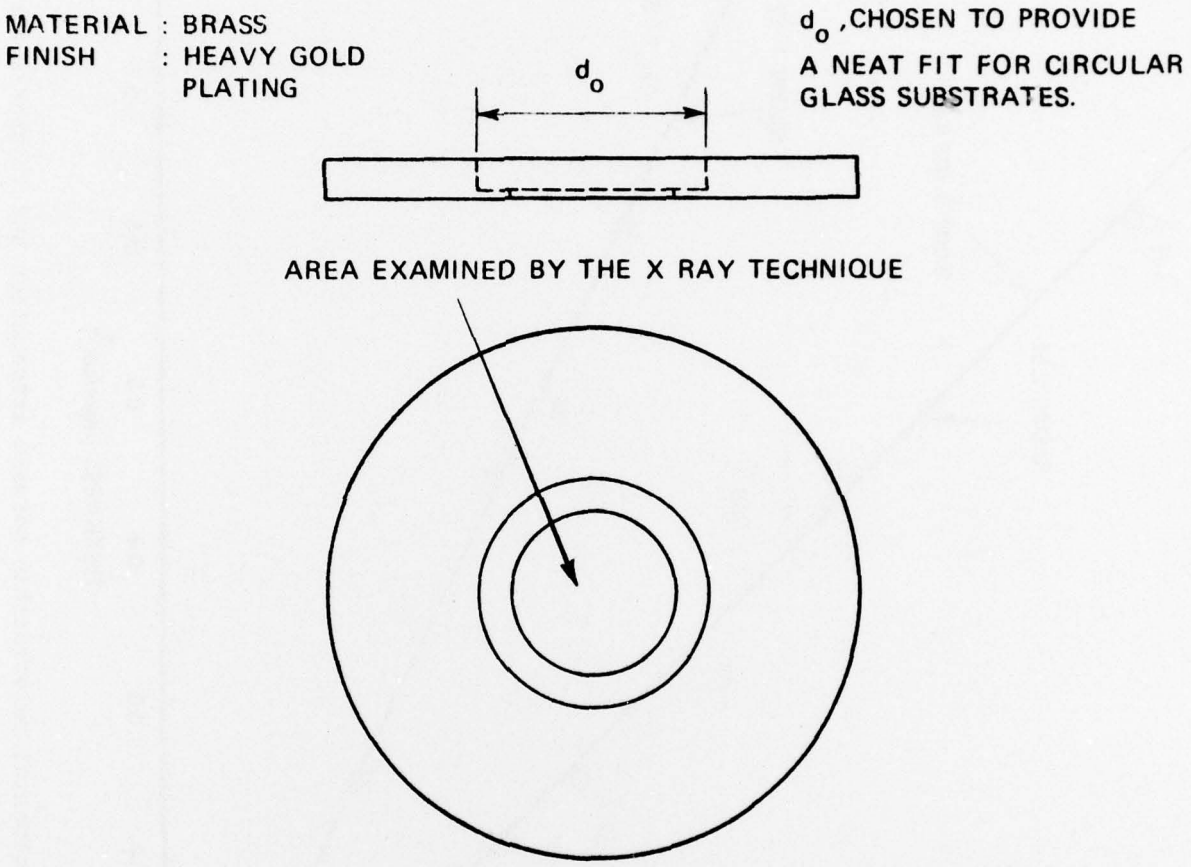

Figure 3. Film holder

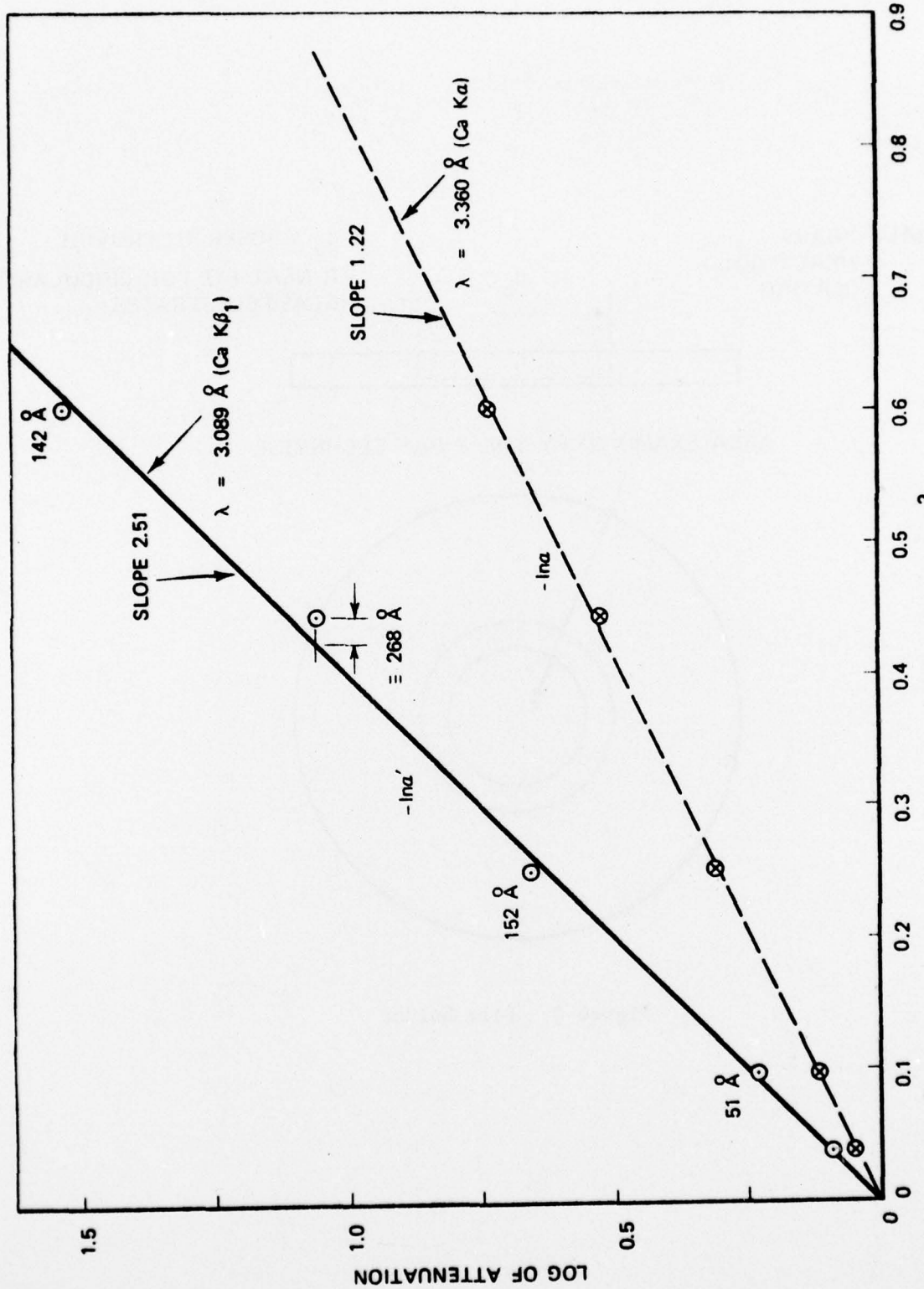

Figure 4. Experimental relationship between attenuation and thickness for Indium films

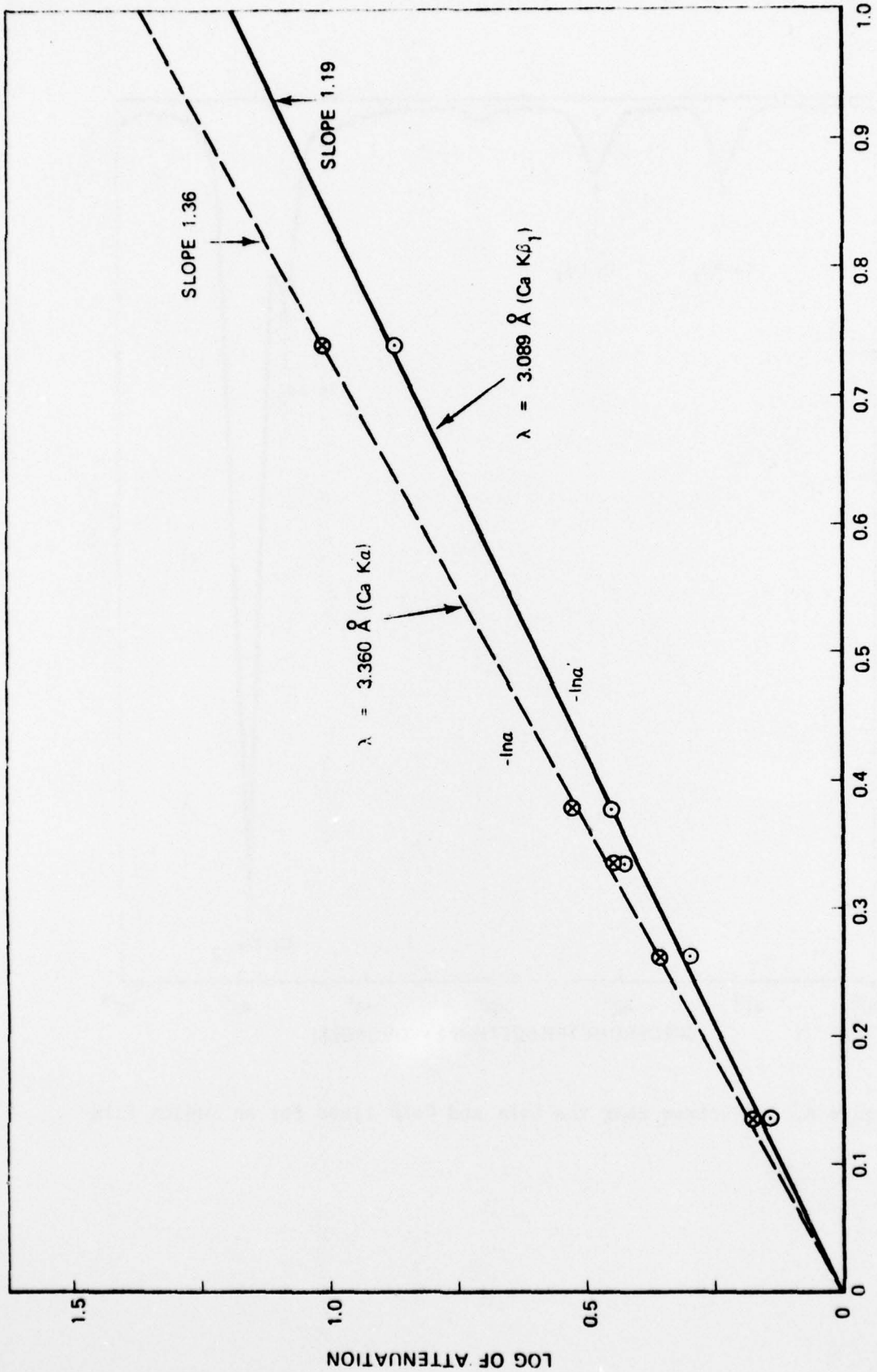

Figure 5. Experimental relationship between attenuation and thickness for Antimony films

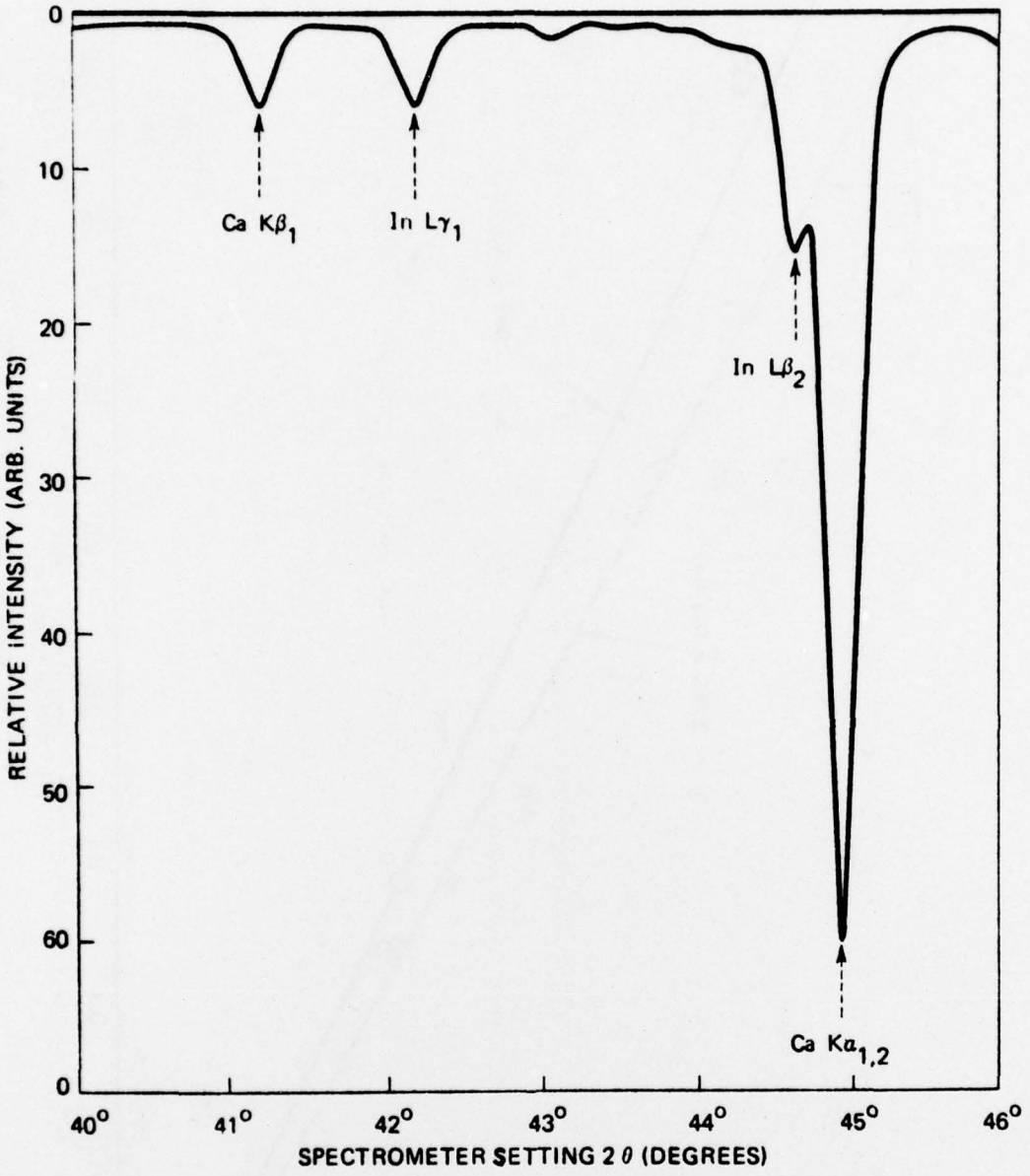

Figure 6. Spectrum near the Cakα and CaKβ lines for an indium film

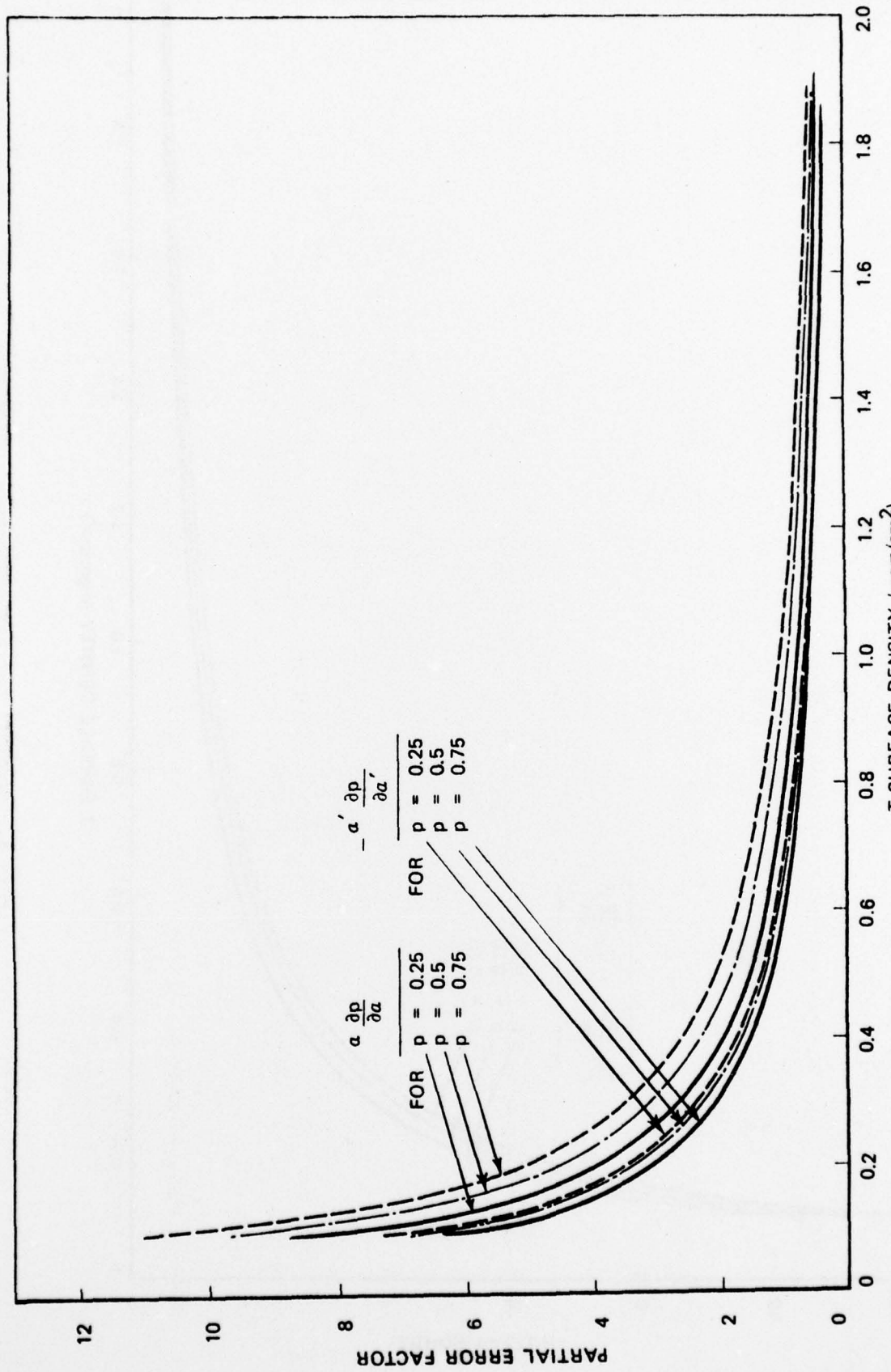

Figure 7. Error components for percentage composition

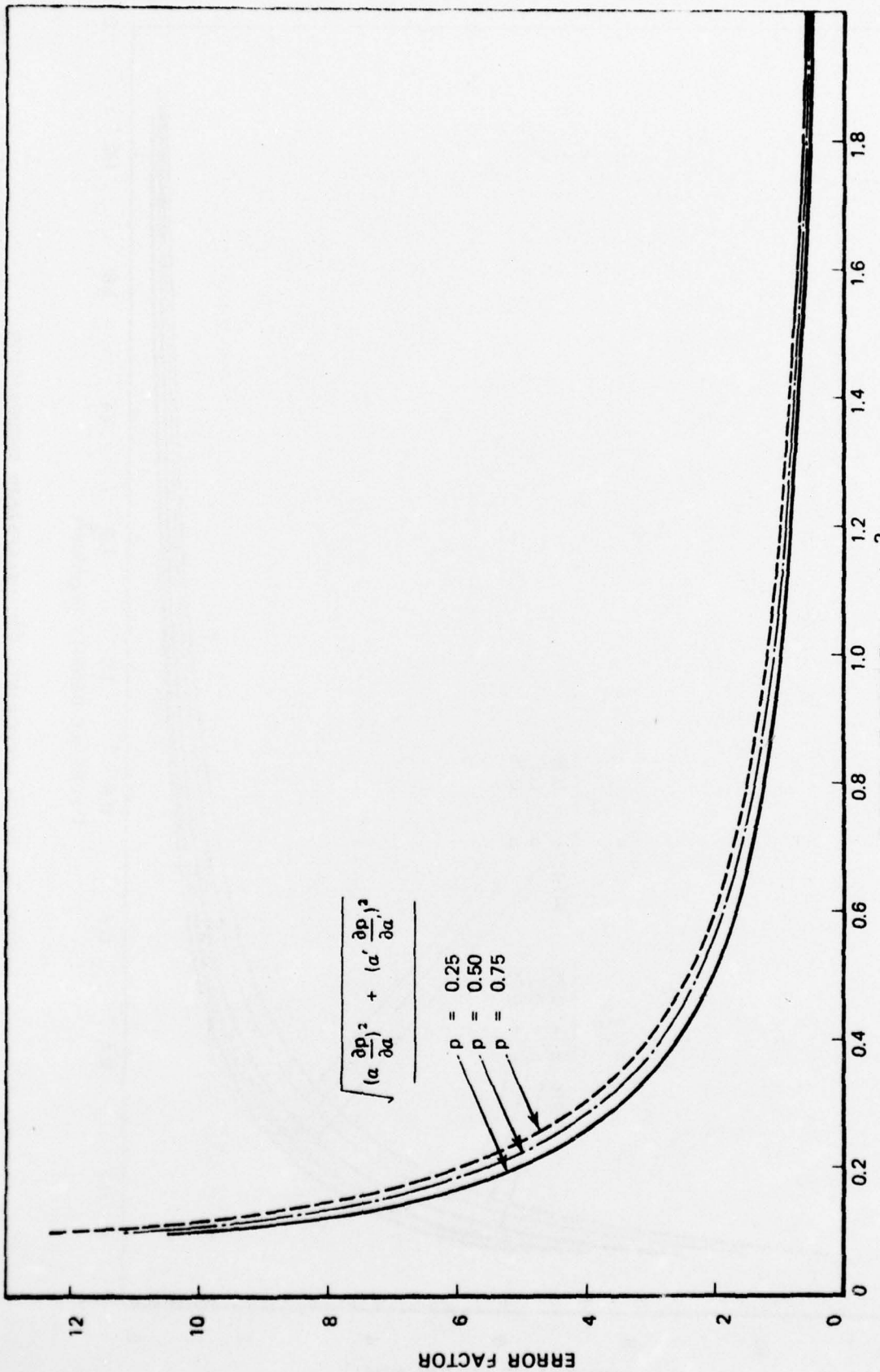

Figure 8. Error factor for percentage composition

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